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- (71) Applicant (*for all designated States except US*): QUEST INTERNATIONAL B.V. [NL/NL]; Huizerstraatweg 28, NL-1411 GP Naarden (NL).
- (72) Inventors; and
- (75) Inventors/Applicants (*for US only*): NESS, Jeremy, Nicholas [GB/GB]; 4 Clevedon Court, Green Lane, Challock, Ashford, Kent, TN25 4BW (GB). GOODALL, Marcus, James [GB/GB]; 9 Hillside Court, Hillside Street, Hythe, Kent CT21 5EH (GB). BEERLING, Judi, Ann [GB/GB]; 32 Linksway, Folkestone, Kent CT19 5LW (GB).
- (74) Agent: KEITH W NASH & CO; 90-92 Regent Street, Cambridge CB2 1DP (GB).

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**WO 03/015736 A2**

(54) Title: PERFUME-CONTAINING COMPOSITION

(57) Abstract: An aqueous skin-care, hair-care or fabric-care composition comprises surfactant, silicone insoluble in water, and perfume having a solubility parameter (SP) not exceeding about 20. By using a perfume with a low SP value, the invention enables good partitioning of perfume into the silicone of the composition, which means that the perfume will be associated with the silicone and deposited therewith onto skin, hair or fabric in use.

Title: Perfume-Containing Composition

Field of the Invention

This invention relates to perfume-containing compositions, particularly skin-care, hair-care and fabric-care compositions.

Background to the Invention

It is well known to use silicones (i.e. silicon polymers made up of -Si-O- linkages known as siloxane units) in compositions for use on the hair and/or skin. Silicones can have conditioning benefits on hair, improving the appearance and feel of hair, and impart soft, smooth properties to the skin. It is also known to use silicones in fabric-care compositions, particularly rinse conditioners, to improve the feel of fabric.

The present invention concerns the performance of perfume in compositions, in terms of behaviour relative to silicone and desirably also deposition of perfume onto hair, skin or fabric and retention during drying.

Summary of the Invention

According to the present invention there is provided an aqueous skin-care, hair-care or fabric-care composition, comprising surfactant, silicone insoluble in water, and perfume having a solubility parameter not exceeding about 20.

The term "solubility parameter" refers to the Hildebrand parameter, which is a measure of cohesion well known in physical chemistry. A full description may be found in the text "Solubility Parameters and other Cohesion Parameters" by A F Barton, published by CRC Press (1983). Solubility parameter (SP) may be determined from equation (1) below, but is more conveniently estimated by a variety of group additive methods. The method due to Hoy (see section 6.4 in the above reference of Barton) has been used in this invention.

**Equation (1)**

$$\text{Solubility Parameter} = [(dH - RT)/V]^{1/2}$$

where:       $dH$     = molar enthalpy of vaporisation  
                 $V$     = molar volume  
                 $R$     = the gas constant  
                 $T$     = temperature

Solubility parameter is a measure that was originally developed as a means of explaining or predicting the likely solubility of particular liquids in one another, particularly the amount of a polymer that will dissolve in a solvent. In the context of the present invention, solubility parameter is used as a measure of the amount of a solvent (perfume) that will dissolve in a polymer (silicone).

If a silicone and perfume have relatively similar solubility parameter values, then it would be expected that the perfume will have good solubility in the silicone.

Silicones generally have very low solubility parameters, usually below 15 and typically in the range 11 to 14. Water has a very high solubility parameter of 47.9. Thus in an aqueous composition including silicone and perfume of relatively close SP values, it would be expected that the perfume will partition into, i.e. dissolve into, the silicone to a reasonable extent.

The solubility parameters of the perfume and silicone are preferably as close as possible. Thus the perfume preferably has a solubility parameter < 19, more preferably < 18 and most preferably < 17.

The solubility behaviour of the perfume is also affected by its chemical nature. In particular, a perfume with strong hydrogen bonding capability, e.g. an alcohol, will preferentially dissolve into the aqueous phase and so partition poorly into the silicone.

Hydrogen bonding ability can be quantified as the hydrogen bonding factor (HBF). HBF as used in this specification is based on work by W. Gordy and S.C. Standford, as detailed in the following publications:

- J. Chem. Phys. (1939) 7 93-99  
J. Chem. Phys. (1940) 8 170-177  
J. Chem. Phys. (1941) 9 204-214

The HBF is expressed in terms of a value representing a spectroscopic shift. The higher the shift the stronger the (hydrogen) bonding and hence high HBF materials are those capable of hydrogen bonding. At the extremes of the scale, alcohols have HBF values of about 222 and hydrocarbons have HBF values of about 57.

It is thus preferred to use a perfume with a relatively low HBF, preferably < 125. In this case the perfume SP should be < 20, preferably < 19 and most preferably < 18. It is further preferred to use a perfume with a lower HBF, e.g. < 115, desirably < 100, e.g. hydrocarbons, esters etc.

Surprisingly we have found that it is possible to use a perfume with a higher HBF (> 125) provided the perfume has a lower SP value. Thus if the perfume has a HBF > 125, the SP should be < 19, preferably < 18, most preferably < 17.

Solubility parameter and hydrogen bonding factor values for a number of typical common perfume materials, categorised on the basis of the above guidance, are given below:

Highly preferred perfume materials, i.e. SP < 19 and HBF < 100

	SP	HBF
Limonene	16.5	57
DMBCA*	18.4	87
Linalyl acetate	16.8	87
Fenchyl acetate	17.1	87
Myrcene	16.4	57
Roseacetone	14.8	81
Manzanate	17.1	87
Yara**	18.9	69
Orange terpenes	16.4	57

\*Dimethyl benzyl carbonyl acetate

\*\*2-methoxy naphthalene

Preferred perfume materials i.e. SP < 19 or HBF < 115

	SP	HBF
Jasmacyclene	19.1	87
Adoxal	17.1	117
Allyl amyl glycollate	17.9	138
Diphenyl methane	19.4	57
delta damascone	19.1	110
Anisole	19.9	69
Musk ketone	21.6	110
Benzyl acetate	20.2	87
Galaxolide	17.5	138

Jasmacyclene is a Trade Mark of Quest, Adoxal is a Trade Mark of Givaudan and Galaxolide is a Trade Mark of IFF.

**Generally unsuitable perfume materials, i.e. SP>19 and HBF>115**

	SP	HBF
Anisic aldehyde	23.1	117
Linalol	19.8	222
Cinnamic aldehyde	22.4	117
Isoeugenol	22.2	222
Nerol	20.3	222
Rastone	22.2	120
Indole	25.9	209
Hexyl salicylate	20.0	120

Rastone is an abbreviation for Raspberry Ketone (4-(4-hydroxyphenyl)-2-butanone).

The perfume will typically comprise a mixture of perfume materials, blended to achieve desired odour properties. The approach used above for single materials has been found to be useful for designing mixtures. In the case of a mixture, values for SP and HBF are calculated as a weighted mean based on the percentages by weight of different components in the mixture. For example, for a mixture of a% by weight of perfume material A with an SP value of SP<sub>a</sub> and b% by weight of perfume material B with an SP value of SP<sub>b</sub>, where a + b = 100%, then the weighted mean SP of the mixture is: -

$$\frac{(a \times SP_a) + (b \times SP_b)}{100}$$

We have found that it is possible to use a mixture of perfume materials including one or more generally unsuitable materials (e.g. included to achieve desired odour properties) provided the SP and HBF values of the mixture as a whole are appropriate. Non-odorous materials, eg diluents, coupling materials (discussed below) should not be included in these calculations.

The perfume is typically present in the composition in an amount in the range 0.01 to 10%, preferably 0.1 to 1%, by weight of the weight of the composition.

By using a perfume with a suitably low SP value, the invention enables good partitioning of perfume into the silicone of the composition, which means that the perfume will be associated with, and carried with, the silicone.

The silicone is insoluble in water. The term insoluble is used in this context to mean that less than 0.01% by weight of the silicone dissolves in water at room temperature (about 20°C) and at typical temperatures of use (about 40°C). The silicone is preferably also substantially insoluble in the surfactant of the composition, with the term substantially insoluble being used in this context to mean that less than 1% by weight of the silicone dissolves in the surfactant at room temperature (about 20°C) and at typical temperatures of use (about 40°C). The insolubility requirements mean that, in preferred embodiments at least, the silicone is generally present in the composition in undissolved form, and so is available for the perfume to partition thereunto.

The composition is preferably designed to enable good deposition of the silicone (and the perfume carried thereby) onto skin, hair or fabric in use of the composition. To this end it is preferred to use certain silicones or mixtures of silicones that have good deposition properties, e.g. as is known in the art, while also allowing perfume to partition thereinto.

Suitable silicone materials having good deposition properties include the following:

1. Silicone elastomers, i.e. crosslinked silicones exhibiting rubber-like elastic properties. Such materials are described in general terms in the article "Silicone Wonders" in the May 2001 issue of the magazine "Global Cosmetic Industry" produced by Cosmix Inc., New York, USA. Silicone elastomers are also disclosed in EP 0240350A, US 5049377, CA 2275845, US 5929162 and US 5969035. The silicone elastomers are preferably in the form of small particles, typically having a particle size <5µm, preferably <2µm. Suitable materials are commercially available from companies including Dow Corning,

Shin-Etsu, Wacker etc. One currently preferred silicone elastomer is a dimethicone/vinyl dimethicone crosspolymer available from Dow Corning under the product designation DC 9506: this is supplied as a dry powder which can be made up into an aqueous slurry (which is a preferred form for use in the invention).

Silicone elastomers not only have good deposition properties, but also allow perfume to partition into them, absorbing the perfume. A silicone elastomer may therefore be used on its own in a composition in accordance with the invention.

2. The silicone elastomer may also be used mixed with a liquid carrier, generally a silicone carrier, into which perfume partitions well. The elastomer and carrier are preferably in the form of an emulsion or dispersion, preferably of small droplet or particle size, preferably  $< 5\mu\text{m}$ , more preferably  $< 2\mu\text{m}$ .

Suitable carriers can be classified into a number of different types as follows:

a) Volatile carriers, e.g. as disclosed in EP 0240350A, including volatile cyclic silicones such as cyclomethicone, volatile linear silicones and volatile hydrocarbons such as isododecane. Commercially available examples of volatile cyclic silicones including the silicone fluids available from Dow Corning under the product designations DC244, DC255, DC344, DC345 and DC246. Commercially available examples of volatile linear silicones include the material available from Dow Corning under the product designation DC200/0.65. Commercially available examples of volatile hydrocarbons include Isopar materials (Isopar is a Trade Mark) available from Exxon.

b) Non-volatile carriers such as phenyl-substituted silicones, i.e. polyphenylmethylsiloxanes. A commercially available example of such a non-volatile carrier is the material available from Dow Corning under the product designation DC 556.

Examples of silicone elastomers and emulsions of dispersions of silicone elastomers in carriers are disclosed, e.g., in US 5969035, US 5929162, CA 2275845 and EP 240350A.

3. A high molecular weight silicone (not being an elastomer) e.g. a silicone gum together with a liquid carrier as described above. While a high molecular weight silicone has good deposition properties, in general it will not allow perfume to partition well thereinto. By use in combination with a carrier into which the perfume partitions well, preferably in the form of an emulsion or dispersion of small droplet size as discussed above, both deposition and partitioning properties are obtained.

In this context, references to a high molecular weight silicone means a silicone having a viscosity of > 350cS, preferably > 10,000cS, more preferably > 100,000cS. Suitable high molecular weight silicones are disclosed, e.g., in EP 0240350A, US 5114706 (column 7), WO00/07551 and WO00/61084. Suitable high molecular weight silicones available commercially, e.g. high molecular weight dimethicones produced by Dow Corning, Union Carbide, General Electric etc. The silicones may have other functionalities (e.g. cationic groups). Mixtures of high molecular weight silicones and carrier are also commercially available, e.g. dimethicone in volatile silicone available from Dow Corning under the product designations DC1401 and DC1501.

The high molecular weight silicone of such mixtures preferably constitutes at least 10% by weight, more preferably at least 20% by weight, of the total weight of the mixture.

Silicone is typically present in the composition in an amount in the range 0.1 to 10%, preferably 0.5 to 3%, by weight of the weight of the composition.

The composition preferably includes a deposition aid, e.g. a cationic deposition aid, to enhance deposition. Suitable materials are known in the art, e.g. as disclosed in US 5990059 and EP 0552024A. One preferred class of materials is cationic guar gum derivatives such as guar hydroxypropyltrimonium chloride (which is commercially available from Rhodia under the Trade Mark Jaguar). Particularly preferred is Jaguar C13S, which has a low degree of substitution of the cationic groups and high viscosity.

The deposition aid is suitably present in an amount in the range 0.001 to 5%, preferably 0.01 to 1%, more preferably 0.02 to 0.5%, by weight of the weight of the composition.

The composition preferably further includes one or more coupling materials having a solubility parameter intermediate that of the silicone and the perfume, e.g. 2 to 3 units higher than that of the silicone. The coupling material is thus soluble in both the perfume and the silicone and hence functions to allow the perfume to be better associated with the silicone. The coupling agent may be a perfume ingredient of low SP, e.g. isopropyl myristate (IPM) which has a SP of 16.5. The volatile hydrocarbons mentioned above as possible carrier liquids typically have SP values of about 14 and are also suitable for this purpose.

The composition includes at least one surfactant which may be selected from anionic, nonionic, cationic, amphoteric and zwitterionic surfactants, or mixtures thereof. Suitable surfactants are well known to those skilled in the art, and a suitable surfactant or mixture of surfactants for any particular composition may be readily selected.

Surfactant is typically present in the composition in an amount in the range 3 to 25%, preferably 15 to 20%, by weight of the weight of the composition.

The composition may take the form of a range of hair-care products, e.g. a hair shampoo, conditioner, styling mousse or gel etc., skin-care products e.g. a shower gel, body wash etc. and fabric-care products, e.g. fabric washing products, fabric conditioners etc. Suitable formulations for the different product types are well known to those skilled in the art.

Optional ingredients suited to the different products types may be included, if desired, in known manner.

In use of the composition, with preferred embodiments at least, the silicone with perfume partitioned therein deposits onto skin, hair or fabric in use, so that perfume is also

effectively deposited and retained during drying, giving improved dry perfume performance on skin/hair/fabric.

In a further aspect, the invention provides a method of enhancing perfume deposition onto skin, hair or fabric from an aqueous skin-care, hair-care or fabric-care composition comprising surfactant, the method comprising adding to the composition silicone insoluble in water and perfume having a solubility parameter not exceeding about 20.

The invention will be further described, by way of illustration in the following Examples and with reference to the accompanying drawings in which:

Figure 1 is a graph of % headspace depression versus solubility parameter.

Example 1

A shampoo composition was made by mixing together the following ingredients:

	<u>weight %</u>
Sodium lauryl ether sulphate 2EO*	45.0
Cocoamidopropyl betaine**	7.0
Euperlan PK810***	10.0
Jaguar C13S****	0.2
Potassium sorbate (preservative)	0.5
Citric acid (pH adjustment)	0.2
Sodium chloride (thickener)	0.7
Silicone	3.0
Perfume	0.5
Water	to 100.0

\*Empicol ESB3 (27% active (aq)) (Empicol ESB3 is a Trade Mark), Albright and Wilson Ltd. (anionic surfactant)

\*\*Tegobetaine L7 (30% active (aq)) (Tegobetaine L7 is a Trade Mark), Th. Goldschmidt AG (amphoteric surfactant)

\*\*\*Euperlan PK810 (Euperlan PK810 is a Trade Mark), Henkel, is a pearlising agent with the INCI designation glycol distearate and fatty alcohol ether sulphate and cocamide MEA and laureth 10.

\*\*\*\*Rhodia

The total surfactant content of the composition is about 16% by weight.

The composition of the perfume was as follows:

Ingredient	wt %	SP	HBF
Adoxal	0.1	17.1	117
Allyl amyl glycollate	0.5	17.9	138
Benzyl acetate	10.0	20.2	87
cis 3 hexenyl salicylate	3.0	20.0	120
Damascone delta	1.0	19.1	110
DMBCA	2.5	18.4	87
Diphenyl methane	0.1	19.4	57
Galaxolide*	30.0	17.5	138
Jasmacyclene	1.0	19.1	87
Linalyl acetate	14.0	16.8	87
Methyl dihydrojasmonate	27.0	19.2	110
Methyl ionone alpha iso	5.0	17.6	110
Orange terpenes	5.8	16.4	57
Weighted Mean		18.2	109.4

\* Galaxolide is a Trade Mark of IFF.

Different formulations were made up using the following silicones (all from Dow Corning):

1. DC9506 elastomer (dimethicone/vinyl dimethicone crosspolymer)
2. Silicone blend DC9040 (DC9506 in cyclomethicone carrier)
3. DC9506 (one part by weight) blended with DC246 volatile cyclic silicone (three parts by weight)

4. DC1401 (high molecular weight silicone gum in cyclomethicone carrier).

Example 2

The following experiment was carried out to illustrate partitioning of perfume into silicone.

A mixture of equal weights of the following five perfume materials was prepared:

<u>Material</u>	<u>SP</u>
Anisic aldehyde	23.1
Jasmacyclene	19.1
Fenchyl acetate	17.1
Adoxal	17.1
DMBCA	18.4

This mixture was then dosed into a shampoo base (the shampoo composition given in Example 1 excluding perfume and silicone) at 0.5wt% to provide a Reference sample, and at 0.5wt% into the same shampoo base also containing 1.0wt% of DC9506 elastomer – to provide a Silicone sample. The samples were then allowed to equilibrate for one week at room temperature. An analysis of the equilibrium perfume headspace above the two samples was then carried out using standard gas chromatographic (GC) techniques. The size of the individual perfume peaks in the two shampoo samples was then compared and a % drop (depression) calculated for the ingredient in the silicone sample compared the reference sample. This depression was assumed to be due to perfume partitioning into the silicone phase and thus no longer contributing to the headspace of the sample. This headspace depression is thus a measure of whether an ingredient partitions into the silicone – it is not however quantitative in that a 20% drop in headspace does not necessarily correlate to a 20% partitioning level into the silicone. In fact, the headspace depression usually is lower than the level of partitioning.

The results are shown in Figure 1, from which it can be seen there is a strong correlation between the headspace depression of each ingredient and its solubility parameter.

Claims

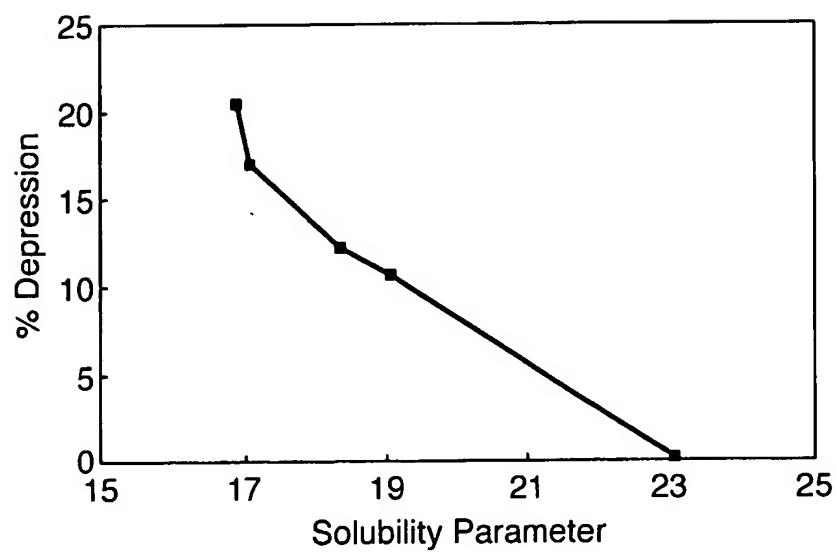
1. An aqueous skin-care, hair-care or fabric-care composition, comprising surfactant, silicone insoluble in water, and perfume having a solubility parameter not exceeding about 20.
2. A composition according to claim 1, wherein the perfume has a solubility parameter (SP) < 19, preferably < 18 and most preferably < 17.
3. A composition according to claim 1 or 2, wherein the perfume has a hydrogen bonding factor (HBF) < 125, preferably < 115, more preferably < 100.
4. A composition according to claim 1, 2 or 3, wherein the perfume has an HBF < 125 and an SP < 20, preferably < 19.
5. A composition according to claim 1, 2 or 3, wherein the perfume has an HBF > 125 and an SP < 19, preferably < 18, most preferably < 17.
6. A composition according to any one of the preceding claims, where the perfume is present in the composition in an amount in the range 0.01 to 10%, preferably 0.1 to 1%, by weight of the weight of the composition.
7. A composition according to any one of the preceding claims, wherein the silicone comprises a silicone elastomer.
8. A composition according to claim 7, wherein the silicone elastomer is mixed with a liquid carrier.
9. A composition according to any one of claims 1 to 6, wherein the silicone comprises a high molecular weight silicone mixed with a liquid carrier.

10. A composition according to claim 9, wherein the high molecular weight silicone constitutes at least 10% by weight, preferably at least 20% by weight, of the total weight of the mixture.
11. A composition according to claim 8, 9 or 10, wherein the silicone and carrier are in the form of an emulsion or dispersion.
12. A composition according to any one of claims 8 to 11, wherein the carrier is selected from: volatile carriers, including volatile cyclic silicones, volatile linear silicones and volatile hydrocarbons; and non-volatile carriers, including phenyl-substituted silicones.
13. A composition according to any one of the preceding claims, wherein silicone is present in the composition in an amount in the range 0.1 to 10%, preferably 0.5 to 3%, by weight of the weight of the composition.
14. A composition according to any one of the preceding claims, further comprising a deposition aid.
15. A composition according to claim 14, wherein the deposition aid comprises a cationic guar gum derivative.
16. A composition according to claim 14 or 15, wherein the deposition aid is present in an amount in the range 0.001 to 5%, preferably 0.01 to 1%, more preferably 0.02 to 0.5%, by weight of the weight of the composition.
17. A composition according to any one of the preceding claims, wherein the composition further includes one or more coupling materials having a solubility parameter intermediate that of the silicone and the perfume.

18. A composition according to any one of the preceding claims, wherein surfactant is present in the composition in an amount in the range 3 to 25%, preferably 15 to 20%, by weight of the weight of the composition.
19. A composition according to any one of the preceding claims, wherein the silicone is substantially insoluble in the surfactant.
20. A composition according to any one of the preceding claims, comprising shampoo.
21. A method of enhancing perfume deposition onto skin, hair or fabric from an aqueous skin-care, hair-care or fabric-care composition comprising surfactant, the method comprising adding to the composition silicone insoluble in water and perfume having a solubility parameter not exceeding about 20.

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Fig.1.



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(75) Inventors/Applicants (*for US only*): NESS, Jeremy, Nicholas [GB/GB]; 4 Clevedon Court, Green Lane, Challock, Ashford, Kent, TN25 4BW (GB). GOODALL, Marcus, James [GB/GB]; 9 Hillside Court, Hillside Street, Hythe, Kent CT21 5EH (GB). BEERLING, Judi, Ann [GB/GB]; 32 Linksway, Folkestone, Kent CT19 5LW (GB).

(74) Agent: KEITH W NASH & CO; 90-92 Regent Street, Cambridge CB2 1DP (GB).

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**WO 03/015736 A3**

(54) Title: PERFUME-CONTAINING COMPOSITION

(57) Abstract: An aqueous skin-care, hair-care or fabric-care composition comprises surfactant, silicone insoluble in water, and perfume having a solubility parameter (SP) not exceeding about 20. By using a perfume with a low SP value, the invention enables good partitioning of perfume into the silicone of the composition, which means that the perfume will be associated with the silicone and deposited therewith onto skin, hair or fabric in use.

## INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER  
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## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
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Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 96 19562 A (PROCTER & GAMBLE (US)) 27 June 1996 (1996-06-27) claims 1-3, 7, 11 example VII	1-6, 13, 18-21
A	page 1, paragraph 2 page 2, paragraph 3 - paragraph 4 page 6, paragraph 2 -page 7, paragraph 1 ---	7-12, 14-17
A	DE 958 046 C (GOLDSCHMIDT AG TH) 14 February 1957 (1957-02-14) claim page 1, line 17 -page 2, line 3 ---	1-21
		-/-

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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- \*A\* document defining the general state of the art which is not considered to be of particular relevance
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Date of the actual completion of the international search

Date of mailing of the International search report

11 April 2003

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Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Neys, P

## INTERNATIONAL SEARCH REPORT

Int'l Application No  
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